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L6: Entry 3 of 12

File: USPT

Jun 5, 2001

DOCUMENT-IDENTIFIER: US 6241779 B1

TITLE: Metal ligand containing bleaching compositions

Brief Summary Text (4):

Desirable qualities for paper include strength, whiteness and brightness. The strength of the paper is related to the viscosity of the pulp employed in its manufacture which, in turn, is related to the condition of the cellulose after the pulping operation. Molecular cellulose, as explained above, is a linear chain of d-glucose which naturally forms long fibers. The longer the individual cellulose polymer chains, the higher the viscosity of the pulp, and in turn, the greater the strength of the paper. Thus, during processing, it is most desirable to avoid cleaving the cellulose polymers into smaller units.

Brief Summary Text (13):

The environmental impacts of wastewaters produced by pulp and paper processing have been the focus of significant research over the past 30 years. The traditional areas of concern have been oxygen demand, suspended solids and acute toxicity. Improvements in control strategies within mills, pulping and bleaching technology, and secondary treatment systems have addressed these issues to a large extent. There is now an increasing focus on potential sub-acute toxicity (e.g. reproductive effects), residual nutrients/eutrophication, and recalcitrant constituents, especially color and organochlorine. Reductions in wastewater color and absorbable organic halogen mass loadings following biological treatment may average 10% and 40%, respectively. In some cases, significant increases in color levels may occur. Approximately 50% of the soluble chemical oxygen demand in bleached kraft mill effluents (BKME) also remains following secondary treatment and appears to consist of recalcitrant high molecular mass material (HMM). The higher molecular mass constituents (MW>1000 Daltons) in BKME consist primarily of highly degraded, chlorinated lignin degradation products with some residual polysaccharide constituents. In BKME, this material may constitute 40-90% of the total organic material, approximately 80% of the AOX content, and 60-100% of the color loadings from the mill. Little information is available on the chemical nature or mass flows of color and HMM discharged from other mill operations (e.g. mechanical pulping).

Detailed Description Text (20):

The conventional hydrogen peroxide bleaching methods are practiced at a pH within the range of 11 to 9 and at temperatures within the range of 30 to 80.degree. C., and most often at 50 to 70.degree.. See, Charles, J. E. et al., 1980 TAPPI Pulping Conference Proceedings, TAPPI Press (1980). When one of the activators of the present invention is used, the temperature of the reaction can be reduced to ambient temperature. While the catalyst activators can be used at the higher conventional reaction temperatures, they also works well at 35 and 40.degree. C. In some applications, a higher temperature may be preferred, for example, a temperature up to about 130.degree. C., and preferable within a range of ambient to 90.degree. C. It is known that, for about every ten degrees in temperature, the reaction rate changes by a factor of about two. Thus, the reaction rate is much faster at higher temperatures. However, when bleaching pulp with the activator of the present invention, rates of H.sub.2 O.sub.2 oxidation which are significantly better than those heretofore possible can be obtained with temperatures much lower than heretofore possible, thereby saving energy costs and increasing plant throughput rates where other features of the mill make this possible. Preferred temperature ranges are therefore between ambient and 130.degree. C., preferably between ambient and 90.degree. C. and most preferably between ambient and 60.degree. C. For some applications, the preferred range is between about ambient and 90.degree. C. The bleaching system of the invention will even function effectively at sub-ambient temperatures. The wide range of temperatures over which the activator will function permits the method of the present invention to be used in existing facilities and in conjunction with other pulp and paper bleaching processes without having to make special temperature adjustments for the peroxide bleaching portion of a commercial production line, other than the normally advantageous change of lowering the

temperature.

Detailed Description Text (43):

Further, suitable amphoteric and zwitterionic surfactants which contain an anionic water-solubilizing group, a cationic group and a hydrophobic organic group may include amino carboxylic acids and their salts, amino dicarboxylic acids and their salts, alkylbetaines, alkyl aminopropylbetaines, sulfobetaines, alkyl imidazolinium derivatives, certain quaternary ammonium compounds, certain quaternary phosphonium compounds and certain tertiary sulfonium compounds.

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L6: Entry 11 of 12

File: USPT

May 14, 1991

DOCUMENT-IDENTIFIER: US 5015412 A

TITLE: Alkaline tolerant sulfobetaine amphoteric surfactantsAbstract Text (1):

Disclosed is an aqueous basic solution having a calculated pH of 13 or greater of a sulfobetaine which is soluble and stable for extended periods of storage. The sulfobetaine also is soluble and stable in aqueous basic solutions of high concentration, e.g. up to 30%-50% by weight sodium hydroxide or potassium hydroxide.

Brief Summary Text (6):

The present invention is directed to a class of surfactants which are soluble in strongly basic alkaline solutions (i.e. a calculated pH of 13 or greater, advantageously 15 or greater), are storage stable for extended time periods of storage in strongly basic alkaline solutions, and are very low to moderate foamers. Moreover, such surfactants retain their surfactant properties when incorporated into strongly basic aqueous solutions. The novel surfactants which possess such unique combination of properties are alkyl sulfobetaines wherein the alkyl group ranges from about 6 to 12 carbon atoms in chain length. Another aspect of the present invention is an aqueous solution (calculated pH of 13 or greater) of the alkyl sulfobetaine and a base, such as, for example, sodium hydroxide or potassium hydroxide in concentrations ranging up to 30%-50% in concentration.

Detailed Description Text (2):

The amphoteric sulfobetaine surfactants of the present invention are unique in their ability to meet diverse criteria required of them in formulating aqueous basic surfactant solutions having a calculated pH of 13 or greater. This high pH requirement of the present invention distinguishes the novel aqueous betaine solutions, for example, from the detergent compositions of U.S. Pat. Nos. 3,539,521 and 3,619,115 which utilize betaines having R.sub.1 being C.sub.12 -C.sub.18 and preferably C.sub.14 -C.sub.16 at pH levels of up to 11.5-12.0. At pH levels above 13, however, the betaines lose water solubility at chain lengths of R.sub.1 of 12 and greater. Thus, the C.sub.6 -C.sub.12 chain length restriction of R.sub.1 in the present invention. Also, the intended industrial cleaning preferred uses of the novel aqueous betaine solution dictate the much higher pH or caustic levels used in the present invention.

Detailed Description Text (3):

The sulfobetaines of the present invention can be represented conventionally by the following general structure: ##STR1## where R is a C.sub.6 -C.sub.12 alkyl group;

Detailed Description Text (6):

Within the alkyl group range of about C.sub.6 -C.sub.12 the sulfobetaine possesses the requisite degree of water solubility and surfactancy required of it. At chain lengths above C.sub.12, water solubility of the sulfobetaine at high pHs becomes a problem and typically is lost (i.e. the sulfobetaine becomes insoluble in highly alkaline water). While various reaction schemes may be envisioned for synthesis of the alkyl sulfobetaines of the present invention, the following two-step reaction scheme currently is favored where R.sub.4 is OH. The initial step involves the formation of an epichlorhydrin/bisulfite intermediate. This reaction conveniently is conducted in water in the presence of a base (for example, sodium hydroxide) at relatively moderate reaction temperatures (e.g. 120.degree.-200.degree. F.) and preferably under inert atmosphere.

Detailed Description Text (7):

Following the formation of the epichlorhydrin/bisulfite intermediate, such intermediate is reacted with the appropriate amine for forming the product sulfobetaine. This second reaction step is conducted at reaction temperatures ranging from about 100.degree. to 200.degree. F. Unreacted material then can be neutralized and/or removed and the pH and percent non-volatile solids of the reaction product adjusted as is necessary, desirable, or convenient in conventional fashion. For the sulfobetaine where R.sub.4 is

H, a propyl sultone, ##STR2## can be reacted with the appropriate amine. The resulting alkyl sulfobetaine is soluble in caustic solution at concentrations ranging up to 10% by weight and greater. Moreover, such solubility is present even at elevated levels of potassium hydroxide, sodium hydroxide, or like bases ranging in concentrations of greater than 30%, advantageously 30%-50%, and preferably 40%-50%. Based upon the definition of pH, a theoretical maximum value of just in excess of about 15.5 is possible. It will be appreciated, though, that discussions of pH become less meaningful at these ultra-high levels of caustic. Also, use of sodium or potassium hydroxide becomes quite preferred in order to reach the foregoing caustic concentration.

Detailed Description Text (8):

A variety of bases may be used in conjunction with the sulfobetaines of the present invention. Such bases include, for example, sodium hydroxide, potassium hydroxide, calcium hydroxide, calcium oxide, sodium metasilicate, tetrapotassium pyrophosphate, sodium tripolyphosphate, trisodium phosphate, potassium silicate, and the like, and even mixtures thereof. As the Examples will demonstrate, the alkyl dimethyl hydroxy sulfobetaines of the present invention are stable in potassium hydroxide and sodium hydroxide solutions ranging up to about 40-50 percent concentration.

Detailed Description Text (9):

The aqueous basic solutions of sulfobetaines of the present invention find use in a variety of applications. Such applications include for example, bottle washing compounds, hot vat cleaning compounds, paper pulping, paint strippers, railroad and aircraft cleaners, dairy and food plant cleaners, detergent sanitizers, polymer-based wax strippers, and the like. The excellent stability, surfactancy, and low foaming characteristics of the alkyl dimethyl sulfobetaine caustic solutions make them useful in these and a variety of additional applications.

Detailed Description Text (13):

Lauryldimethyl sulfobetaine (R.sub.4 =OH) was made by a two-step process described herein. The first step involved the charging of a small Parr reactor with sodium bisulfite (242 g), epichlorohydrin (228 g), deionized water (910 g), and solid sodium hydroxide (2 g). The water and base were mixed and nitrogen sparged to remove dissolved oxygen prior to charging the reactor. The reactor was pressurized to 20 psi with nitrogen and heated to 125.degree. F. at which point the reaction exothermed to a reaction temperature of 140.degree.-150.degree. F. The reaction was conducted for one hour and then sampled for determination of free sodium bisulfite. After the one hour reaction time, this analysis showed that the percent free sodium bisulfite was 0.2 percent. The reactor was cooled to 100.degree. F. and the product removed as the reaction was judged to be complete.

Detailed Description Text (14):

1310 g of the thus-formed intermediate then was added to a three liter-four neck flask along with 416 g of lauryldimethyl amine. The flask was heated to 150.degree.-160.degree. F. and maintained at this temperature while the contents in the flask were stirred. After six hours reaction time, the contents in the flask changed from a milky liquid to a clear liquid. The reaction was continued for a total of 18 hours at which point the reaction was judged to be essentially complete. Sodium hydroxide (18 g, 50% concentration) was added to the flask and the temperature increased to 180.degree. F. to hydrolyze unreacted epichlorohydrin/bisulfite intermediate. After two hours reaction time, the flask again was sampled and analyzed for percent free NaCl which proved to be 8.0 percent. The contents of the flask then were cooled to 100.degree. F. and sufficient sulfuric acid (25% concentration) was added to adjust the pH to about 8-8.5. The final analysis of the lauryldimethyl hydroxy sulfobetaine is set forth below:

Detailed Description Text (15):

The lauryldimethyl hydroxy sulfobetaine was tested for solubility in aqueous potassium hydroxide solution. Concentrations of potassium hydroxide at 10%, 20%, 30%, 40%, and 50% solutions were formulated at percent solids content of lauryldimethyl hydroxy sulfobetaine of 1%, 3%, 5%, and 10%. The lauryldimethyl hydroxy sulfobetaine was judged to be soluble at all concentrations of sulfobetaine at all concentrations of potassium hydroxide. The lauryldimethyl hydroxy sulfobetaine then was subjected to Ross-Miles foam test at 1.0% by weight actives in 72.degree. F. distilled water. The following foam heights were measured: initial, 205 mm; and +5 minutes, 26 mm. In 150 ppm hard (Ca) water at 1% concentration, Ross-Miles foam heights were: initial, 200 mm; and +5 minutes, 29 mm. Thus, it will be seen that the lauryldimethyl hydroxy sulfobetaine is low foaming as well as soluble in high concentrations of potassium hydroxide.

Detailed Description Text (16):

Next, the stability of the lauryldimethyl sulfobetaine to alkaline solutions was evaluated. Initial samples of the lauryldimethyl hydroxy sulfobetaine at 1%, 3%, and 5% by weight of a 50% solids solution of the sulfobetaine were established for 40% sodium hydroxide solutions. Surface tension and interfacial tension (against refined mineral oil, Nujol oil) were recorded initially, after one month storage in polyethylene bottles, and after 6 months of storage in polyethylene bottles. Samples for the tension evaluation were prepared by diluting the concentrate to 3% sodium hydroxide in deionized water for taking the measurements. The following results were recorded:

Detailed Description Text (17):

The above-tabulated results clearly demonstrate that the lauryldimethyl hydroxy sulfobetaine remains virtually unaffected when stored for time periods of up to six months in concentrated sodium hydroxide solutions. Thus, the lauryldimethyl hydroxy sulfobetaine has been demonstrated to be soluble in concentrated alkaline solutions, storage stable in concentrated alkaline solutions, and low foaming.

Detailed Description Text (19):

An octyl dimethyl hydroxy sulfobetaine was made in a manner like that described in Example 1. At 5% by weight sulfobetaine, Ross-Miles foam heights in deionized water were: initial, 47 mm; and +5 minutes, 40 mm. In 150 ppm (Ca) hard water, Ross-Miles foam heights were: initial, 43 mm; and +5 minutes, 36 mm. The low foaming property of this betaine is demonstrated.

Detailed Description Text (20):

Samples of the octyl dimethyl hydroxy sulfobetaine were compounded at 1%, 0.1%, 0.025%, and 0.01% solids in deionized water for tension measurements. The following results were recorded.

Detailed Description Text (21):

These results clearly demonstrate the excellent surfactancy of the octyl dimethyl hydroxy sulfobetaine

Detailed Description Text (22):

Solubility of the octyl dimethyl hydroxy sulfobetaine in the alkaline solutions was evaluated by dissolving the surfactant into a 50% NaOH solution at active levels of 1%, 3% and 5%. The following results were obtained:

Detailed Description Text (23):

Again, the novelty of the inventive sulfobetaines in high (pH of 13 or greater) caustic aqueous solutions is demonstrated.

Detailed Description Paragraph Table (2):

Detailed Description Paragraph Table (2):																	Surface				
Tension and Interfacial Tension Measurements (Dynes/cm) Lauryldimethyl Initial One																					
Month Storage Six Month Storage Sulfobetaine Surface Interfacial Surface Interfacial																					
Surface Interfacial (% weight)* Tension Tension Tension Tension Tension Tension																					
																	0	55.4	16.5		
55.6	16.8	58.0	--	1	40.8	12.4	34.7	12.5	36.2	16.2	3	26.5	9.0	23.5	4.7	27.5	7.2	5	24.0		
6.8	22.4	5.1	26.1	6.8																	
																	*% by weight				
sulfobetaine of a 50% solids solution of the																	sulfobetaine, 40% NaOH, which was stored				
and then diluted to 3% NaOH for these tests.																					

Detailed Description Paragraph Table (3):

Detailed Description Paragraph Table (57)										Surface Tension and Interfacial Tension				
Measurements (Dynes/cm) Octyl Dimethyl Hydroxy Sulfobetaine (% solids)										Surface Tension				
Interfacial Tension										0.01	57.7	30.5	0.025	45.5
19.3	0.1	28.9	6.4	1.0	23.8	2.4								

## CLAIMS:

1. A method for making a storage stable aqueous basic solution having a calculated pH of 13 or greater of a sulfobetaine and the following general structure: ##STR3## where R.sub.1 is a C.sub.6 -C.sub.8 alkyl group,

R.sub.2 and R.sub.3 are CH.sub.3, 2-hydroxy ethyl or 2-hydroxy propyl, which comprises:

- (a) forming an aqueous epichlorohydrin/bisulfite intermediate;
  - (b) reacting said intermediate and a C.sub.6 -C.sub.12 alkyl, R.sub.2, R.sub.3 amine in an aqueous reaction mixture; and
  - (c) adding sodium hydroxide to the thus-formed aqueous solution of said sulfo betaine in an amount of at least 50% by weight if not already present therein to achieve said calculated pH of greater than 13.
3. The method of claim 1 wherein the proportion of said sulfo betaine and said aqueous basic solution ranges from between about 0.05 and 10 percent by weight.

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L6: Entry 8 of 12

File: USPT

Dec 29, 1998

DOCUMENT-IDENTIFIER: US 5853428 A

TITLE: Metal ligand containing bleaching compositions

Brief Summary Text (7):

Desirable qualities for paper include strength, whiteness and brightness. The strength of the paper is related to the viscosity of the pulp employed in its manufacture which, in turn, is related to the condition of the cellulose after the pulping operation. Molecular cellulose, as explained above, is a linear chain of d-glucose which naturally forms long fibers. The longer the individual cellulose fibers, the higher the viscosity of the pulp, and in turn, the greater the strength of the paper. Thus, during processing, it is most desirable to avoid cleaving the cellulose polymers into smaller units.

Detailed Description Text (16):

The conventional hydrogen peroxide bleaching methods are practiced at a pH within the range of 11 to 9 and at temperatures within the range of 30.degree. to 80.degree. C., and most often at 50.degree. to 70.degree. C. See, Charles, J. E. et al., 1980 TAPPI Pulping Conference Proceedings, TAPPI Press (1980). When the activator of the present invention is used, the temperature of the reaction can be reduced to ambient temperature. While the catalyst activator can be used at the higher conventional reaction temperatures, it also works well at 35.degree. and 40.degree. C. It is known that, for about every ten degrees in temperature, the reaction rate changes by a factor of about two. Thus, the reaction rate is much faster at higher temperatures. However, when bleaching pulp with the activator of the present invention, rates of H.sub.2 O.sub.2 oxidation which are significantly better than those heretofore possible can be obtained with temperatures much lower than heretofore possible, thereby saving energy costs and increasing plant throughput rates. Preferred temperature ranges are therefore between ambient and 80.degree. C., preferably between ambient and 70.degree. C. and most preferably between ambient and 40.degree. C. The bleaching system of the invention will even function effectively at sub-ambient temperatures. The wide range of temperatures over which the catalyst activator will function permits the method of the present invention to be used in existing facilities and in conjunction with other pulp and paper bleaching processes without having to make special temperature adjustments for the peroxide bleaching portion of a commercial production line, other than the advantageous change of lowering the temperature.

Detailed Description Text (37):

Further, suitable amphoteric and zwitterionic surfactants which contain an anionic water-solubilizing group, a cationic group and a hydrophobic organic group may include amino carboxylic acids and their salts, amino dicarboxylic acids and their salts, alkylbetaines, alkyl aminopropylbetaines, sulfobetaines, alkyl imidazolinium derivatives, certain quaternary ammonium compounds, certain quaternary phosphonium compounds and certain tertiary sulfonium compounds. Other examples of potentially suitable zwitterionic surfactants can be found described in Jones, U.S. Pat. No. 4,005,029, at columns 11-15, which are incorporated herein by reference.

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L5: Entry 45 of 59

File: USPT

Nov 11, 1997

DOCUMENT-IDENTIFIER: US 5686014 A

TITLE: Bleach compositions comprising manganese-containing bleach catalysts

Brief Summary Text (4):

Metal-containing catalysts have been described in bleach compositions, including manganese-containing catalysts such as those described in EP 549,271; EP 549,272; EP 458,397; U.S. Pat. No. 5,244,594; U.S. Pat. No. 5,246,621; EP 458,398; U.S. Pat. No. 5,194,416; and U.S. Pat. No. 5,114,611. These bleach catalysts are described as being active for catalyzing the bleaching action of peroxy compounds against various stains. Several of these bleaching systems are said to be effective for use in washing and bleaching of substrates, including laundry and hard surfaces (such as machine dishwashing, general cleaning) and in the textile, paper and wood pulp industries.

Detailed Description Text (86):

Detergent Surfactants--Nonlimiting examples of surfactants useful herein typically at levels from about 1% to about 55%, by weight, include the conventional C.sub.11 -C.sub.18 alkyl benzene sulfonates ("LAS") and primary, branched-chain and random C.sub.10 -C.sub.20 alkyl sulfates ("AS"), the C.sub.10 -C.sub.18 secondary (2,3) alkyl sulfates of the formula CH.sub.3 (CH.sub.2).sub.x (CHOSO.sub.3.sup.- M.sup.+)CH.sub.3 and CH.sub.3 (CH.sub.2).sub.y (CHOSO.sub.3.sup.- M.sup.+) CH.sub.2 CH.sub.3 where x and (y+1) are integers of at least about 7, preferably at least about 9, and M is a water-solubilizing cation, especially sodium, unsaturated sulfates such as oleyl sulfate, the C.sub.10 -C.sub.18 alkyl alkoxy sulfates ("AE.sub.x S"; especially EO 1-7 ethoxy sulfates), C.sub.10 -C.sub.18 alkyl alkoxy carboxylates (especially the EO 1-5 ethoxycarboxylates), the C.sub.10-18 glycerol ethers, the C.sub.10 -C.sub.18 alkyl polyglycosides and their corresponding sulfated polyglycosides, and C.sub.12 -C.sub.18 alpha-sulfonated fatty acid esters. If desired, the conventional nonionic and amphoteric surfactants such as the C.sub.12 -C.sub.18 alkyl ethoxylates ("AE") including the so-called narrow peaked alkyl ethoxylates and C.sub.6 -C.sub.12 alkyl phenol alkoxyates (especially ethoxylates and mixed ethoxy/propoxy), C.sub.12 -C.sub.18 betaines and sulfobetaines ("sultaines"), C.sub.10 -C.sub.18 amine oxides, and the like, can also be included in the overall compositions. The C.sub.10 -C.sub.18 N-alkyl polyhydroxy fatty acid amides can also be used. Typical examples include the C.sub.12 -C.sub.18 N-methylglucamides. See WO 9,206,154. Other sugar-derived surfactants include the N-alkoxy polyhydroxy fatty acid amides, such as C.sub.10 -C.sub.18 N-(3-methoxypropyl) glucamide. The N-propyl through N-hexyl C.sub.12 -C.sub.18 glucamides can be used for low sudsing. C.sub.10 -C.sub.20 conventional soaps may also be used. If high sudsing is desired, the branched-chain C.sub.10 -C.sub.16 soaps may be used. Mixtures of anionic and nonionic surfactants are especially useful. Other conventional useful surfactants are listed in standard texts.

Detailed Description Text (142):

Other Ingredients--A wide variety of other ingredients useful in detergent compositions can be included in the compositions herein, including other active ingredients, carriers, hydrotropes, processing aids, dyes or pigments, solvents for liquid formulations, solid fillers for bar compositions, etc. If high sudsing is desired, suds boosters such as the C.sub.10 -C.sub.16 alkanolamides can be incorporated into the compositions, typically at 1%-10% levels. The C.sub.10 -C.sub.14 monoethanol and diethanol amides illustrate a typical class of such suds boosters. Use of such suds boosters with high sudsing adjunct surfactants such as the amine oxides, betaines and sultaines noted above is also advantageous. If desired, soluble magnesium salts such as MgCl.sub.2, MgSO.sub.4, and the like, can be added at levels of, typically, 0.1%-2%, to provide additional suds and to enhance grease removal performance.



**End of Result Set****Generate Collection**

L4: Entry 2 of 2

File: USPT

Oct 3, 2000

DOCUMENT-IDENTIFIER: US 6127494 A

TITLE: Method for producing polymers using micellar polymerization

Brief Summary Text (44):

Examples of amphoteric or zwitterionic surfactants which may be selected include betaines, sultaines, glycines, amphoteric imidazoline derivatives and aminopropionates.

Detailed Description Text (54):

Similarly, shredded newsprint is defibered in a Kitchen-Aid mixer with an anchor style beater with the magazine pulp from above and is added to the mixer. Any pulping chemicals are normally added at this point in the process. A water bath is used to keep the Kitchen Aid bowl at a constant temperature.

Detailed Description Text (79):

Pulping Chemicals are as follows: